

## **General Disclaimer**

### **One or more of the Following Statements may affect this Document**

- This document has been reproduced from the best copy furnished by the organizational source. It is being released in the interest of making available as much information as possible.
- This document may contain data, which exceeds the sheet parameters. It was furnished in this condition by the organizational source and is the best copy available.
- This document may contain tone-on-tone or color graphs, charts and/or pictures, which have been reproduced in black and white.
- This document is paginated as submitted by the original source.
- Portions of this document are not fully legible due to the historical nature of some of the material. However, it is the best reproduction available from the original submission.



FACILITY FORM 602

<b>N69-36226</b>	
(ACCESSION NUMBER)	(THRU)
<b>41</b>	<b>1</b>
(PAGES)	(CODE)
<b>CR-105641</b>	<b>03</b>
(NASA CR OR TMX OR AD NUMBER)	(CATEGORY)

**TEXAS INSTRUMENTS**  
**INCORPORATED**



HEAT STERILIZABLE Ni-Cd BATTERY DEVELOPMENT

Jet Propulsion Laboratory  
Contract No. 951972, Modification No. 3

Report for Fifth Quarter  
1 July to 30 September 1968

by

J. P. Elder - Member Technical Staff  
R. L. Crawford - Acting Project Manager

TEXAS INSTRUMENTS INCORPORATED  
Research and Development Laboratories  
Attleboro, Massachusetts

This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, sponsored by the National Aeronautics and Space Administration under Contract NAS-7-100; Task Order No. RD-26.



NOTICE

This report was prepared as an account of government-sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA

- (a) makes warranty of representation, expressed or implied with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- (b) assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employees or contractor of NASA, or employee of such contractor, prepares, disseminates, or provides access to any information pursuant to his employment with such contractor.

Request for copies of this report should be referred to:

National Aeronautics and Space Administration  
Office of Scientific and Technical Information  
Attention: AFSS-A



TABLE OF CONTENTS

List of Tables . . . . . ii

List of Figures . . . . . iii

Abstract . . . . . iv, v

Introduction . . . . . 1

Electrochemical Investigations . . . . . 3

~~Ni-Cd~~ Cells and Component Compression Studies . . . . . 14



### LIST OF TABLES

No.		Page
1	Ni-Cd, $2^4$ Factorial Design Description	3
2	Ni-Cd, $2^4$ Factorial Design Experiment, Replicate Cell Cycling History	7
3	Limited Charge Acceptance Behavior of Cycled, Sterilized Cells	8
4	Ni-Cd, Modified $2^5$ Factorial Design Description	10
5	Ni-Cd, Modified $2^5$ Factorial Design Experiment, Calculation and Significance Testing of Factor Effects	11
6	Cell Number Description for Figures 1 - 12	13
7	Summary of Results of Cell Components Compression Studies	15
8	Separator-Electrode Compression Interaction Data	15
9	Calculated Core Thicknesses for 17, 18 and 19 Plate Configurations	17
10	Ni-Cd, 18 Plate Cell Pre- and Post-Sterilization Cycling Capacity Dat	20
11	Ni-Cd, 18 Plate Cell Pre- and Post-Sterilization Cycling E.C.V. Data	21



---

LIST OF FIGURES

No.		Page
1	17 Plate Factorial Cell, AH Capacity vs Cycle No. Cells #17 and #19	22
2	17 Plate Factorial Cell, AH Capacity vs Cycle No. Cells #25 and #27	23
3	17 Plate Factorial Cell, AH Capacity vs Cycle No. Cells #21 and #23	24
4	17 Plate Factorial Cell, AH Capacity vs Cycle No. Cells #29 and #31	25
5	17 Plate Factorial Cell, E.C.V. vs Cycle No. Cells #17 and #19	26
6	17 Plate Factorial Cell, E.C.V. vs Cycle No. Cells #25 and #27	27
7	17 Plate Factorial Cell, E.C.V. vs Cycle No. Cells #21 and #23	28
8	17 Plate Factorial Cell, E.C.V. vs Cycle No. Cells #29 and #31	29
9	17 Plate Factorial Cell, E.C.R. vs Cycle No. Cells #17 and #19	30
10	17 Plate Factorial Cell, E.C.R. vs Cycle No. Cells #25 and #27	31
11	17 Plate Factorial Cell, E.C.R. vs Cycle No. Cells #21 and #23	32
12	17 Plate Factorial Cell, E.C.R. vs Cycle No. Cells #29 and #31	33



### ABSTRACT

The objective of this work is the development of heat-sterilizable, hermetically sealed Ni-Cd cells for space applications.

The electrochemical characterization of the sterilized factorial design cells is continuing. Data up to and including cycle #30 is presented.

The majority of cells which have failed, employ the #14019 polypropylene separator. Selected data from the original  $2^4$  factorial design has been employed to set up a  $2^5$  factorial design. An analysis of variance of the calculated factor effects has shown that the dominant factor influencing the operation of the Ni-Cd cells is the heat sterilization. However, the concentration and the amount of electrolyte do significantly affect the electrochemical characteristics. The two main characteristics of a heat sterilized cell are (1) an increase of up to 20% in the overall efficiency, above that monitored prior to sterilization, (2) an increase of the order 60 mv in the magnitude of the cell terminal voltage at end-of-charge. Even though the cycling data does show random scatter, there are indications that (1) the efficiency, which does not show any degradation, approaches a limiting value of 74-78% of theoretical on continued cycling, (2) the high E.C.V., exhibited immediately following sterilization, is a permanent characteristic.





The initial results of a compression study of cell components have indicated that the #FT2140 polypropylene separator is more suitable than the #14019 separator. Further, calculations based on these findings have indicated that an 18-plate configuration is more suitable than a 17-plate arrangement. Initial pre- and post-sterilization cycling studies of such 18-plate cells tend to bear out this conclusion. These cells exhibit the two main sterilization characteristics, viz, high end-of-charge voltage and increased efficiency. Furthermore, the cells appear to show greater reproducibility than the 17-plate configuration cells.



## 1. Introduction

This is the fifth quarterly progress report on the heat-sterilizable nickel-cadmium battery development under Jet Propulsion Laboratory Contract No. 951972, Modification No. 3, sponsored under NASA Contract NAS-7-100, Task Order No. RD-26. The object of this contract is to perform research and development work leading to the design, development, fabrication and testing of sealed, rechargeable, nickel-cadmium cells capable of heat-sterilization.

The heat sterilization requirements include testing at 135°C for type approval and at 125°C for flight acceptance. At the 135°C sterilization temperature, the heating rate is 19°C/hour. The chamber is cooled at the same rate at which it is heated. Two such cycles are required. For preliminary testing, one 120-hour cycle may be used.

Since the fourth quarterly report, four new specific tasks have been added to the original contract. Essentially, however, these several tasks may be incorporated into one of three broad categories: (1) Electrochemistry, involving statistical and other experiments aimed at characterizing and optimizing electrodes, electrolyte and separators for heat-sterilizable Ni-Cd cells, (2) Case and Seal Design for hermetically sealed, rechargeable, heat-sterilizable cells, (3) Fabrication - Performance - Cycle Life Testing necessary for the evaluation of 4 AH sealed, rectangular cells before and after sterilization.



The emphasis now is to study and interpret the characteristic electro-chemical behavior of sterilized cells, with a view of optimizing cell design and thereby achieving cell to cell uniformity and reproducibility. The work performed during the fifth quarter is reported herein. Due to the sudden illness of Dr. P. V. Popat, Dr. J. P. Elder has been assigned full time to the project and will assume certain of Dr. Popat's responsibilities.



2. Electrochemical Investigations

2.1 Ni-Cd Cell 2<sup>n</sup> Factorial Experiment

The purpose of the 2<sup>n</sup> factorial experiment is to elucidate the effects of various factors on the electrochemical behavior of the heat sterilizable, hermetically sealed, rechargeable Ni-Cd cell. Originally, a four factor experiment was planned and initiated. The factors chosen, their letter designations and the two levels are shown in Table 1.

TABLE 1

Ni-Cd Rectangular, 17 Plate Cells (8 Positive - 9 Negative).

Theoretical Capacity (formation), 4.96 AH

Factors		Factor Levels	
Designation	Description	Low (0)	High (1)
A	Type of Polypropylene Separator	#14019	#FT2140
B	Concentration of KOH Electrolyte	30 w/o	34 w/o
C	Percentage Pore Fill with KOH Electrolyte	70	80
D	Heat Treatment	Unsterilized	Sterilized

In this particular experiment, because of the choice of the fourth factor, viz, heat treatment, only 8 (2<sup>3</sup>) cells are required per factorial set, each member containing the appropriate combination of



the first three factors, A, B and C. They are first tested prior to sterilization, factor D at the low (0) level. Following sterilization, factor D at the high (1) level, testing is continued. In this manner, one obtains the necessary 16 ( $2^4$ ) sets of data required for the complete calculation of primary and interaction effects. Four replicates of each of the eight cells were constructed. Of the four groups, only the first were fitted with pressure gauges. In the tests performed to date, two charging routines have been employed. In all cases, the cells were discharged at the C/2.5 rate, i.e. with a current of 2.0A and a theoretical capacity of 4.96 AH, based upon the positive plate formation capacity. The first charge routine, R1, is charge at 0.4A for 17 hours, i.e. C/12.5 rate to 137% charge level. The second routine, R2, is a charge at 1.0A for 5 hours, i.e. C/5 rate to 100% charge level. The results for the pre-sterilization cycling tests have been presented in the Fourth Quarterly Report, April 1 - June 30, 1968. During this quarter, all the cells were sterilized. Two groups, #2 and #4, were not cycled. Groups #1 and #3 were cycled, initially for the first six cycles with the R1 charging routine, then, for the remaining cycles, with the R2 routine. Certain effects were observed. Firstly, during charge, the cells exhibited greater internal pressures than had been observed prior to sterilization. For the group 1 cells, the excessive pressure actuated the pressure level switches before the planned charge input level was attained. Secondly, those cells utilizing the 14019 polypropylene separator at a certain cycling stage failed to accept any charge, presumably because of the development of



internal short circuits. The complete charge-discharge history of the four groups of replicate cells up to 9/30/68 is shown in Table 2.

The zero or limited charge acceptance behavior of the cells, marked with an asterisk, is summarized in Table 3. As can be seen, cells 9 and 5 (Group 1), and 3 (Group 3), early lost their ability to accept charge. Other cells, e.g. 27 (Group 1), having exhibited an inability to accept charge, showed a temporary recovery, but later lost this ability again. One important fact emerges from this data. Those cells which show the greatest likelihood of (a) exhibiting excessive cell pressures, and/or (b) losing their ability to accept charge, all utilize the 14019 polypropylene separator. In actuality, later data, which will be reported on in the sixth quarterly report, has shown that all the cells employing this separator, except #13, eventually lose their charge acceptance ability.

In attempting to make maximum use of the existing pertinent data, it is necessary to modify the factorial design. It is known that the magnitudes of the various electrochemical parameters, characterizing the operation of a sealed, sintered plate Ni-Cd cell, are dependent upon the charge-discharge conditions. Therefore, it is important to select pre-sterilization and post-sterilization data obtained under identical charging conditions. This limits the choice to data selected from cycles 1 - 6 (cf. Table 2). Since first and second cycle data is very often atypical, and since there is missing data in cycles 4 and 5, we



are limited to employing cycle 3 and 6 data. Since, of necessity, factor A has had to be removed from the original factorial design, there are two choices available for the analysis of existing data. The data obtained for cycles 3 and 6, and for cells with and without pressure gauges, may be employed in two ways.

- (1) As replicate data to be used in  $2^3$  factorial.
- (2) As two new factors, to be used with the remaining three, B, C and D, to form a  $2^5$  factorial.

Both methods have been employed. The second method has proved more successful, and these results will be reported. The manner in which the two new factors have been incorporated into the  $2^5$  factorial design is shown in Table 4. This modification assumes that any third or fourth order interaction effects are not significant and that any such calculated effects are, in fact, due to experimental error.

The complete results of the analysis of variance for the 32 separate cell data, Efficiency (%), End-of-Charge Voltage (E.C.V.), End-of-Charge Resistance (E.C.R.) and End-of-Discharge Resistance (E.D.R.) are shown in Table 5. E is the calculated effect. The sign preceding the E value indicates the direction of the effect. The error variance is given by the mean square of the effects for the six factor combinations marked with an asterisk. If the effect is numerically greater than the error variance, then the variance ratio, V, is calculated.

TABLE 2

Ni-Cd Rectangular, 17 Plate Cells, Theoretical Capacity (Formation) 4.96 AH. 2<sup>4</sup> Factorial Experiment

Replicate Cells Cycling History to 9/30/68

Factor Code	REPLICATE GROUP 1			REPLICATE GROUP 2		REPLICATE GROUP 3		REPLICATE GROUP 4	
	Cell #	No. Complete Cycles and Charge Routine	Cell #	No. Complete Cycles and Charge Routine	Cell #	No. Complete Cycles and Charge Routine	Cell #	No. Complete Cycles and Charge Routine	Cell #
0 0 0 0	1	37 R1	2	37 R1	3	24 R1	4	24 R1	4
1 0 0 0	17	37 R1	18	37 R1	19	24 R1	20	24 R1	20
0 1 0 0	9	37 R1	10	37 R1	11	24 R1	12	24 R1	12
1 1 0 0	25	37 R1	26	37 R1	27	24 R1	28	24 R1	28
0 0 1 0	5	37 R1	6	37 R1	7	24 R1	8	24 R1	8
1 0 1 0	21	37 R1	22	37 R1	23	24 R1	24	24 R1	24
0 1 1 0	13	37 R1	14	37 R1	15	24 R1	16	24 R1	16
1 1 1 0	29	37 R1	30	37 R1	31	24 R1	32	24 R1	32
0 0 0 1	1	6 R1 24 R2	2	Not Cycled	3*	4 R1 1 R2	4	Not Cycled	4
1 0 0 1	17*	3 R1 11 R2	18	Not Cycled	19	6 R1 25 R2	20	Not Cycled	20
0 1 0 1	9*	1 R1	10	Not Cycled	11*	1 R1	12	Not Cycled	12
1 1 0 1	25*	6 R1 19 R2	26	Not Cycled	27*	6 R1 9 R2	28	Not Cycled	28
0 0 1 1	5*	6 R1	6	Not Cycled	7*	3 R1 1 R2	8	Not Cycled	8
1 0 1 1	21*	6 R1 11 R2	22	Not Cycled	23	6 R1 25 R2	24	Not Cycled	24
0 1 1 1	13	6 R1 24 R2	14	Not Cycled	15*	1 R1	16	Not Cycled	16
1 1 1 1	29	6 R1 24 R2	30	Not Cycled	31	6 R1 25 R2	32	Not Cycled	32





TABLE 3

LIMITED OR ZERO CHARGE ACCEPTANCE BEHAVIOR OF CYCLED,  
STERILIZED GROUPS 1 & 3 CELLS

Cell #	Charge Acceptance Behavior	Cause	No. of Cycles of Occurrence	Cycle Number of Occurrence
17	Charge Interrupted	Excessive Pressure	16	4-7, 10-21
25	Charge Interrupted	Excessive Pressure	5	11-15
21	Charge Interrupted	Excessive Pressure	13	7, 10-21
9	No Charge Accept.	Probably Shorting	30	1-30
5	No Charge Accept.	Probably Shorting	24	7-30
3	No Charge Accept.	Probably Shorting	26	6-31
11	No Charge Accept.	Uncertain	30	1-3, 5-31
27	No Charge Accept.	Uncertain	12	15, 20, 22-31
7	No Charge Accept.	Uncertain	27	2, 4, 6, 8-31
15	No Charge Accept.	Uncertain	30	1-3, 5-31



F indicates the level of significance of the effect, calculated from the statistical F-test. Effects with an F value less than 0.90 (i.e. 10% confidence level) are not considered significant. The large amount of scatter in the resistance data is mirrored in the high error variances. Thus, no great reliance should be placed on the results of the analysis for this data.

## 2.2 Consideration of Factor Effects

### 2.2.1 Cell Efficiency

The efficiency is primarily affected by the three first factors, A, B and C. The effect of the pressure gauge attachment, factor D, is barely significant. The cell overall efficiency is lowered by increasing the electrolyte concentration from 30 w/o to 34 w/o, as shown by the significant (0.999 level), negative effect of factor A. Raising the amount of available electrolyte in the cell results in enhanced efficiency as shown by the significant (0.981 level), positive effect of factor B. The sterilization heat treatment is very effective in increasing efficiency as shown by the significant (0.9999 level), positive effect of factor C. Although no great emphasis should be placed on the significance levels of the first and second order interaction effects, they at least can be employed in ranking the relative importance of the three primary factor effects. Heat sterilization is the dominant factor. Although the evidence is not decisive, it would appear that the percentage pore fill factor ranks second.



TABLE 4

Ni-Cd, RECTANGULAR, 17 PLATE CELL MODIFIED  $2^4$  ( $2^5$ ) FACTORIAL

FACTORS		FACTOR LEVELS	
Designation	Description	Low (0)	High (1)
A	Concentration of KOH Electrolyte	30 w/o	34 w/o
B	% Pore Fill with Electrolyte	70	80
C	Heat Treatment	Unsterilized	Sterilized
D	Pressure Gauge Attachment	with	without
E	Cycle Number	3	6

TABLE

Ni-Cd, RECTANGULAR, 17 PLATE CELL MODIFIED 2<sup>4</sup> (2<sup>5</sup>)

Cell #	A B C D E	Effcy. (%)	E	V	F	E.C.V. (v)	E	V	F
17	0 0 0 0 0	51.1				1.378			
25	1 0 0 0 0	52.8	-6.13	61.97	0.999	1.408	-0.012	4.87	0.930
21	0 1 0 0 0	75.9	2.49	10.22	0.981	1.427	0.003		
29	1 1 0 0 0	40.0	-2.15	7.64	0.967	1.330	-0.0059	1.10	0.665
17	0 0 1 0 0	73.9	13.89	318.6	0.999	1.467	0.056	98.75	0.999
25	1 0 1 0 0	65.6	0.63			1.457	0.0006		
21	0 1 1 0 0	70.2	-1.66	4.57	0.924	1.465	0.0022		
29	1 1 1 0 0	66.5	8.75	126.5	0.999	1.459	0.0149	7.03	0.962
19	0 0 0 1 0	51.7	2.00	6.61	0.958	1.420	0.0109	3.76	0.899
27	1 0 0 1 0	56.1	3.74	23.1	0.997	1.415	0.003		
23	0 1 0 1 0	55.8	-0.18			1.422	0.0049		
31	1 1 0 1 0	53.7	5.09	42.8	0.999	1.413	0.0107	3.67	0.896
19	0 0 1 1 0	79.3	0.70			1.476	-0.0046		
27	1 0 1 1 0	62.9	-3.66	22.2	0.997	1.461	-0.0040		
23	0 1 1 1 0	70.9	1.78	5.2	0.937	1.478	0.0046		
31*	1 1 1 1 0	75.3	-0.56			1.480	-0.0060	1.14	0.674
17	0 0 0 0 1	49.7	-0.73			1.404	0.0017		
25	1 0 0 0 1	52.8	0.86	1.23	0.690	1.409	0.0014		
21	0 1 0 0 1	70.2	0.62			1.431	0.0040		
29	1 1 0 0 1	44.7	0.19			1.413	0.0079	1.97	0.790
17	0 0 1 0 1	70.2	-2.05	6.94	0.961	1.473	-0.0105	3.50	0.890
25	1 0 1 0 1	63.2	-0.36			1.453	-0.0059	1.10	0.665
21	0 1 1 0 1	68.2	-0.1			1.458	-0.0040		
29*	1 1 1 0 1	64.9	0.06			1.451	-0.0041		
19	0 0 0 1 1	52.1	0.79	1.02	0.649	1.412	-0.0109	3.76	0.899
27	1 0 0 1 1	59.5	-0.82	1.12	0.670	1.413	-0.0040		
23	0 1 0 1 1	62.9	0.26			1.420	-0.0014		
31*	1 1 0 1 1	55.8	-0.83	1.12	0.670	1.409	-0.0062	1.24	0.692
19	0 0 1 1 1	75.9	-1.14	2.14	0.806	1.469	0.0054		
27*	1 0 1 1 1	59.2	0.90	1.34	0.709	1.431	0.0023		
23*	0 1 1 1 1	66.9	-0.36			1.469	0.0056	1.00	0.645
31*	1 1 1 1 1	73.9	1.30	2.79	0.854	1.469	0.0077	1.91	0.784

AVGE.      62.2

1.436

ERR. VAR.

4.84

0.00025

FOLDOUT FRAME /

FACTORIAL. C.R. C/12, C.L. 137%, D.R. C/2.5

C.C.R. (mΩ)	E	V	F	E.D.R. (mΩ)	E	V	F	SIGNIFICANT EFFECTS	
								Primary	Interaction
8.83				7.59					
8.84	0.12			8.02	0.83			A	
7.77	-0.009			6.68	0.33			B	
7.20	1.34	4.92	0.932	7.07	1.91	4.09	0.911		AB
9.44	1.40	5.34	0.940	10.96	2.53	7.18	0.963	C	
8.20	0.72	1.44	0.724	9.34	1.08	1.30	0.702		
7.51	0.86	2.04	0.797	7.65	1.09	1.34	0.708		
6.90	1.72	8.12	0.971	7.27	2.28	5.79	0.947		ABC
8.11	0.70	1.32	0.706	7.98	1.24	1.71	0.761	D	
7.40	0.65	1.15	0.675	7.34	1.03	1.19	0.683		AD
8.16	1.59	6.93	0.961	7.96	2.23	5.58	0.944		BD
6.55	1.29	4.53	0.923	6.41	1.73	3.36	0.883		ABD
9.07	1.29	4.57	0.924	9.69	1.45	2.34	0.823		
6.67	1.16	3.68	0.897	6.87	1.83	3.73	0.898		ACD
7.55	1.09	3.26	0.879	7.69	1.89	4.00	0.908		
15.04	1.43	5.61	0.944	15.68	1.90	4.06	0.909		
8.46	0.21			8.16	0.93				
8.75	0.08			9.21	0.61				
7.25	-0.02			7.24	0.50				
7.17	0.21			7.57	0.53				
9.97	0.46			10.80	0.52				CE
8.60	-0.04			9.42	0.51				
7.55	-0.03			7.60	0.56				
6.93	0.22			7.19	0.65				
7.72	0.21			7.91	0.61				
7.26	0.00			7.54	0.51				
7.98	0.09			8.16	0.60				
6.31	0.17			6.56	0.64				
10.77	0.21			10.74	0.90				
6.92	0.07			7.45	0.55				
7.80	0.07			8.19	0.48				
17.19	0.23			25.39	0.60				

8.43

8.85

2.93

7.15

FOLDOUT FRAME

2



### 2.2.2 Cell End-of-Charge Voltage

Only one factor emerges as being significant in affecting the cell terminal voltage at end-of-charge. The sterilization heat treatment results in a significantly higher E.C.V., factor C, positive effect, significant at the 0.9999 level. There is some indication that an increase in the electrolyte concentration from 30 w/o to 34 w/o potassium hydroxide causes a reduction in the end-of-charge voltage but the effect is only significant at the 0.93 level. The dominant sterilization factor is again in evidence in the second order ABC interaction effect.

### 2.3 Cycling Studies on Selected Cells from Factorial Design

As previously indicated, following sterilization, the replicate group 1 and 3 cells have now completed thirty charge-discharge cycles. In the first six cycles, the cells were charged at the C/12.5 rate to 137% charge level. In the remaining cycles, the charge rate was increased to C/5 and the charge level reduced to 100%. The electrochemical capacity, end-of-charge voltage and end-of-charge resistance data, pertaining to all cells employing the FT2140 polypropylene separator, is shown in diagrammatic form in Figures 1 - 12. For convenience, the cell description, cell parameter and appropriate figure number are summarized in Table 6.



TABLE 6

Cell #	[KOH] (w/o)	Pore Fill (%)	Press. Gauge Attachment	Capacity Fig. #	E.C.V. Fig. #	E.C.R. Fig. #
17	30	70	yes	1	5	9
19	30	70	no	1	5	9
25	34	70	yes	2	6	10
27	34	70	no	2	6	10
21	30	80	yes	3	7	11
23	30	80	no	3	7	11
29	34	80	yes	4	8	12
31	34	80	no	4	8	12

As can be seen, these cell parameters exhibited a considerable amount of scatter from cycle to cycle. It is to be noted that the random scatter in the end-of-charge voltage data is due partly to the random scatter in the end-of-charge resistance values, e.g. cell #31. However, this partial correlation cannot always be made, especially when the cell has an attached pressure gauge, e.g. cell #17. Even in the presence of this scatter, certain general trends are indicated on continued cycling:

- (1) The delivered electrochemical capacity, which is significantly increased following sterilization, increases, tending to a limiting value indicative of relatively high efficiency in the range 74-78%.
- (2) Disregarding the random scatter in the resistance data of cells #17 and 31, there appears to be a slight, but gradual, increase in the E.C.R.



- (3) Neglecting voltage data pertaining to cells #17, 25, 21 and 29, which on certain cycles (see Table 3) are not fully charged because of the development of excessive internal pressures, the high end-of-charge voltage persists. It must be pointed out that this increase in E.C.V. is a true effect and not due to an increased "IR" contribution.

At the present time, the dominant sterilization effect together with the random scatter precludes any definitive statement regarding the relative effects of the electrolyte concentration and total amount available at the thirty cycle mark.

### 3.1 Ni-Cd Cell and Component Compression Studies

Ni-Cd cells employing 19 plates, i.e. 10 negatives and 9 positives yield an electrochemical capacity comparable with that obtained with 17 plate cells. However, there is a marked lack of reproducibility. This has been associated with the excessive core compression, which affects electrolyte distribution, resulting in a variable utilization of available active material. A series of core and core component compression studies was initiated in August 1968. For convenience, a summary of the results of the initial studies, reported in the August and September 1968 monthly reports, are presented again. The compression tests were performed on the Instron tensile machine.





TABLE 7

SUMMARY OF RESULTS OF CELL COMPONENTS COMPRESSION STUDIES

Component	Type	Component Thicknesses (mils)						
		Under Compression at					After Compression at	
		50 psi	500 psi	1000 psi	200 psi	1000 psi	%Δ t	
		t	t	%Δ t	t	%Δ t	t	t
Separator	Nylon -Pellon	5.6	3.7	34	3.6	2.7	8.0	7.7
Separator	Polypropylene-14019	5.0	3.8	24	3.6	5.3	8.3	7.8
Sepatator	Polypropylene-FT2140	6.6	4.9	25.8	4.6	6.1	7.6	7.4
Electrode	Positive	26.0	25.0	3.8	24.8	0.8		25.4
Electrode	Negative	27.2	26.4	2.9	26.0	1.5		27.0

TABLE 8

SEPARATOR-ELECTRODE PLATE INTERACTION UNDER 1000 PSI COMPRESSION

Electrode-Separator Combination	Thickness (ins.)		
	Calculated*	Measured	Difference
5 Positive - 4 'Pellon'	0.158	0.153	-0.005 (-3.2%)
5 Negative - 4 'Pellon'	0.166	0.159	-0.007 (-4.2%)
5 Positive - 4 '14019'	0.158	0.143	-0.014 (-8.9%)
5 Negative - 4 '14019'	0.166	0.155	-0.011 (-6.6%)
5 Positive - 4 'FT2140'	0.157	0.149	-0.008 (-5.1%)
5 Negative - 4 'FT2140'	0.164	0.158	-0.007 (-4.2%)

\* Calculated from single component compression studies (see Table 7).



### 3.1.1 Ni-Cd-Cell Component Compressive Characteristics

Five 1 square inch samples of each of the cell components, separated by steel blocks, were tested together. Each test was repeated twice for each component. Three types of separator were examined, Pellon (nylon) as a control, and 14019 and FT2140 (polypropylene). The average values of the fifteen samples, during and after compression, are shown in Table 7.

It is observed, for example in the case of the Pellon separator, an increase in pressure from 50 to 500 psi effects a 34% reduction in thickness but a further increase from 500 to 1000 psi only effects a further thickness reduction of 2.7%. When the thicknesses are measured after compression at 200 and 1000 psi, only a 3.8% permanent reduction in thickness results. As can be seen, of the three separators studies, the permanent reduction in thickness is least (2.6%) for the FT2140 as against 6% for the 14019.

When electrodes and separators in contact are compressed, an interaction is observed. In Table 8 are summarized the results from a series of compression studies on a simulated cell electrode-separator interface, consisting of 5 electrode plates interspersed with 4 separator layers. The calculated pack thicknesses



take into account the permanent reduction in thicknesses of the individual components after compressing at 1000 psi. This data indicates that to a large extent both separator and electrode behave elastically in the compression range 50 - 1000 psi. Thus, under moderate pressure, there is negligible permanent reduction in the total cell core thickness. The lower electrode-separator interaction shown by FT2140 points to this material as the more suitable choice. Using the 1000 psi compression data, one may calculate the total core thicknesses for 17, 18 and 19 plate cells, as shown in Table 9.

TABLE 9

Component	Thickness (mils)	17 Plate		18 Plate		19 Plate	
		No.	Thickness (ins.)	No.	Thickness (ins.)	No.	Thickness (ins.)
Positive	25.4	8	0.203	8	0.203	9	0.229
Negative	27.0	9	0.243	10	0.270	10	0.270
FT2140	7.0	18	0.133	19	0.141	20	0.148
Interface	-1.0	34	-0.034	36	-0.036	38	-0.038
Total			0.545		0.578		0.609

These figures show that the 18 plate configuration is the closest to the inner dimension of the cell case, viz. 0.585". The 17-plate cells, 40 mils under, could very well be so loosely packed that the separator is not in sufficiently intimate contact with



the electrodes for complete utilization of available active material. On the other hand, the 19 plate cores, 24 mils over, may require excessive compression to fit into the case. This could result in permanent mechanical damage to one or more of the several components during assembly and further mechanical damage induced by the excessive compression resulting from sterilization. With these points in mind, a number of 18 plate cells were assembled and filled to the 80% pore fill level with 30% potassium hydroxide. The assembled cells were divided into three groups and studied in the following manner.

Group A. Subjected to 5 charge-discharge cycles, routine R1.

Group B. Subjected to 5 charge-discharge cycles, routine R1 then sterilized and re-subjected to 5 similar cycles.

Group C. Sterilized then subjected to 5 charge-discharge cycles., routine R1.

The complete data is shown in Table 10 (delivered capacity) and Table 11 (end-of-charge voltage). As can be seen, the behavior shows similar characteristics to that shown by the 17 plate cells. On pre-sterilization, after the first atypical cycle, commonly observed in Ni-Cd cells, both the capacity and the end-of-charge voltage stabilize. Following sterilization, there is a 25-30% increase in capacity and an increase in E.C.V. of the order 60 mv. The characteristic post-sterilization behavior appears to be



independent of the pre-sterilization history of the cells, although there is a slight indication that the post-sterilization E.C.V. is higher in cells which have undergone pre-sterilization charge-discharge cycles. This initial data indicates that, in comparison with 17 plate cells, 18 plate cells exhibit greater reproducibility.

Cycling studies on both the sterilized, factorial design, 17 plate cells, and the 18 plate cells are continuing. The results of these extended studies will be reported in the sixth quarterly report.



TABLE 10

Ni-Cd 18 PLATE RECTANGULAR CELLS, 30% KOH, 80% PORE FILL,  
FT2140 SEPARATOR

C.R. C/12.5 C.L. 137% D.R. C/2.5 CAPACITY DATA (AH)

Group	Pre-Sterilization						Post-Sterilization				
	Cell #	Cycle #1	Cycle #2	Cycle #3	Cycle #4	Cycle #5	Cycle #1	Cycle #2	Cycle #3	Cycle #4	Cycle #5
A	15	3.134	2.784	2.466	2.634	2.652					
A	16	3.418	3.052	2.784	2.900	2.918					
A	17	3.034	2.652	2.334	2.518	2.584					
B	1	3.400	3.200	3.000	2.784	2.866	4.052	4.018	3.934	4.039	3.952
B	2	3.584	3.300	3.018	2.784	2.834	4.018	3.952	3.818	3.900	3.752
B	3	3.600	3.318	3.052	2.834	2.918	3.952	3.918	3.834	3.952	3.866
B	5	3.418	3.152	2.884	2.634	2.766	4.084	4.034	3.966	4.118	4.052
C	9						4.018	4.052	3.818	3.900	3.866
C	10						4.100	4.184	4.000	3.984	3.918
C	11						4.152	4.200	4.118	4.052	3.966
Average		3.369	3.065	2.791	2.727	2.809	4.061	4.051	3.927	4.009	3.910
Stan. Dev.		0.214	0.256	0.285	0.134	0.131	0.058	0.107	0.112	0.081	0.095



TABLE 11

Ni-Cd 18 PLATE RECTANGULAR CELLS, 30% KOH, 80% PORE FILL, FT2140 SEPARATOR  
C.R. C/12.5 C.L. 137% D.R. C/2.5 END OF CHARGE VOLTAGE DATA (volts)

Group	Pre-Sterilization						Post-Sterilization				
	Cell #	Cycle #1	Cycle #2	Cycle #3	Cycle #4	Cycle #5	Cycle #1	Cycle #2	Cycle #3	Cycle #4	Cycle #5
A	15	1.423	1.410	1.415	1.405	1.411					
A	16	1.423	1.412	1.416	1.407	1.414					
A	17	1.416	1.405	1.409	1.400	1.406					
B	1	1.447	1.420	1.409	1.415	1.402	1.489	1.506	1.474	1.473	1.472
B	2	1.445	1.418	1.409	1.413	1.404	1.488	1.499	1.470	1.468	1.467
B	3	1.446	1.418	1.410	1.414	1.405	1.488	1.509	1.478	1.477	1.479
B	5	1.443	1.416	1.409	1.413	1.404	1.487	1.505	1.475	1.476	1.477
C	9						1.486	1.475	1.473	1.460	1.462
C	10						1.487	1.472	1.471	1.460	1.465
C	11						1.484	1.472	1.471	1.460	1.465
Average		1.435	1.413	1.411	1.416	1.406	1.487	1.488	1.474	1.468	1.468
Stan. Dev.		0.0134	0.0059	0.0031	0.0056	0.0043	0.0017	0.016	0.0026	0.0026	0.0076

FIG. 1

AMPERE CAPACITY OF 17 PLATE FACTORIAL CELLS  
VERSUS  
CYCLE NUMBER

CELL DESIGN:

TYPE FT2140 SEPARATOR  
30 % KOH 70 % PORE FILL

STERILIZED

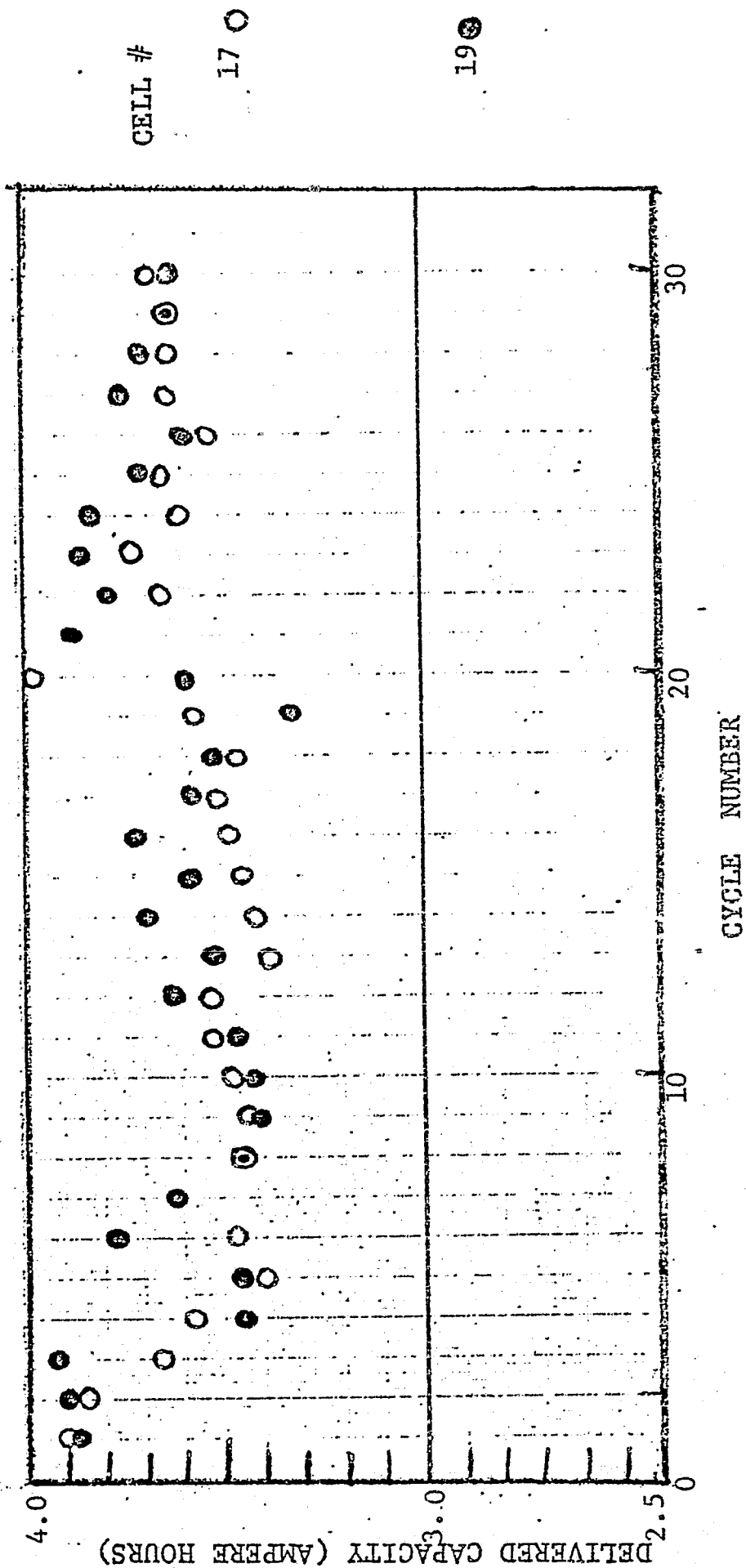




FIG. 2

AMPERE CAPACITY OF 17 PLATE FACTORIAL CELLS  
VERSUS  
CYCLE NUMBER

CELL DESIGN:

TYPE FT2140 SEPARATOR  
34% KOH 70% FORE FILL

STERILIZED

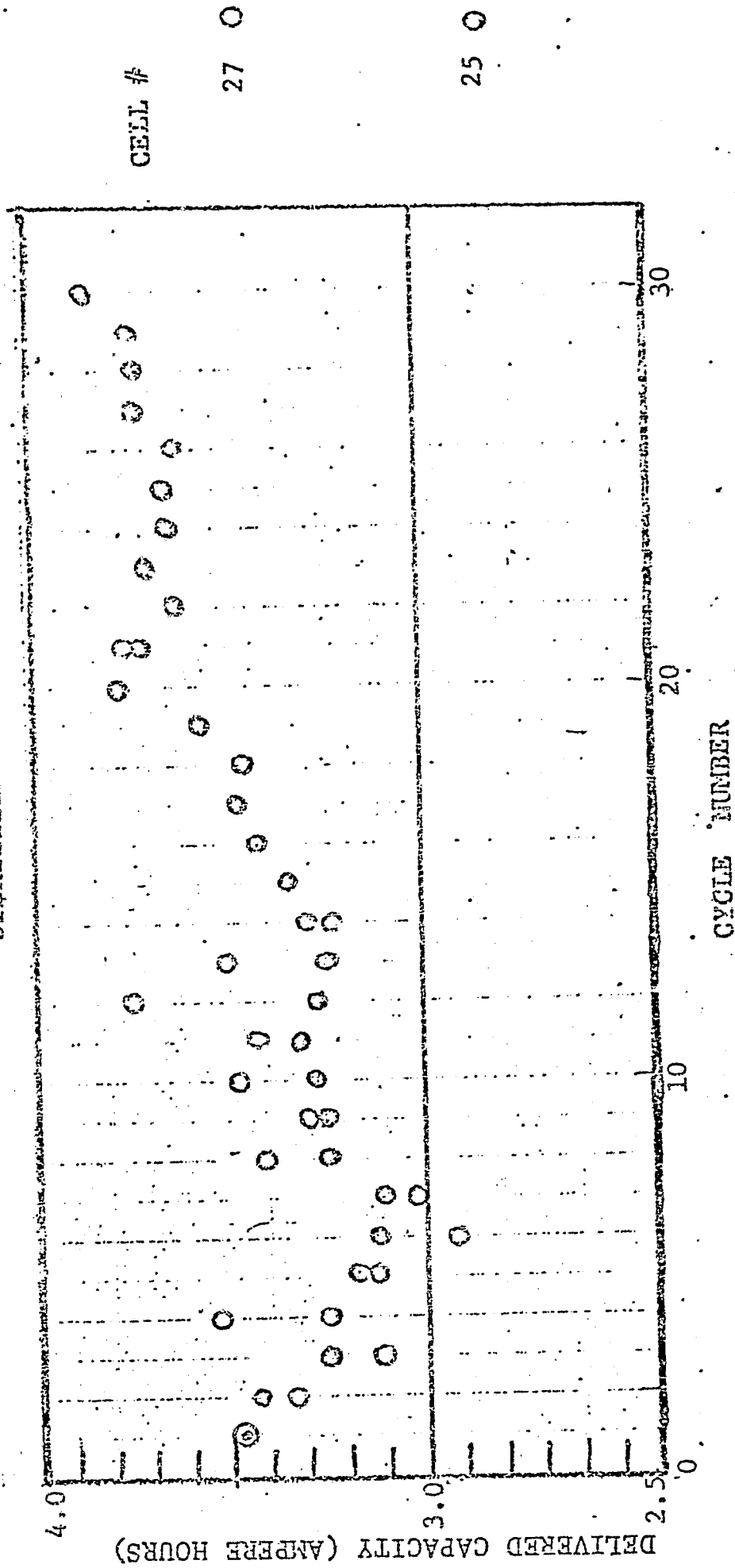


FIG. 3

AMPERE CAPACITY OF 17 PLATE FACTORIAL CELLS  
VERSUS  
CYCLE NUMBER

CELL DESIGN:

TYPE FT2140 SEPARATOR  
30 % KOH 80 % PORE FILL

STERILIZED

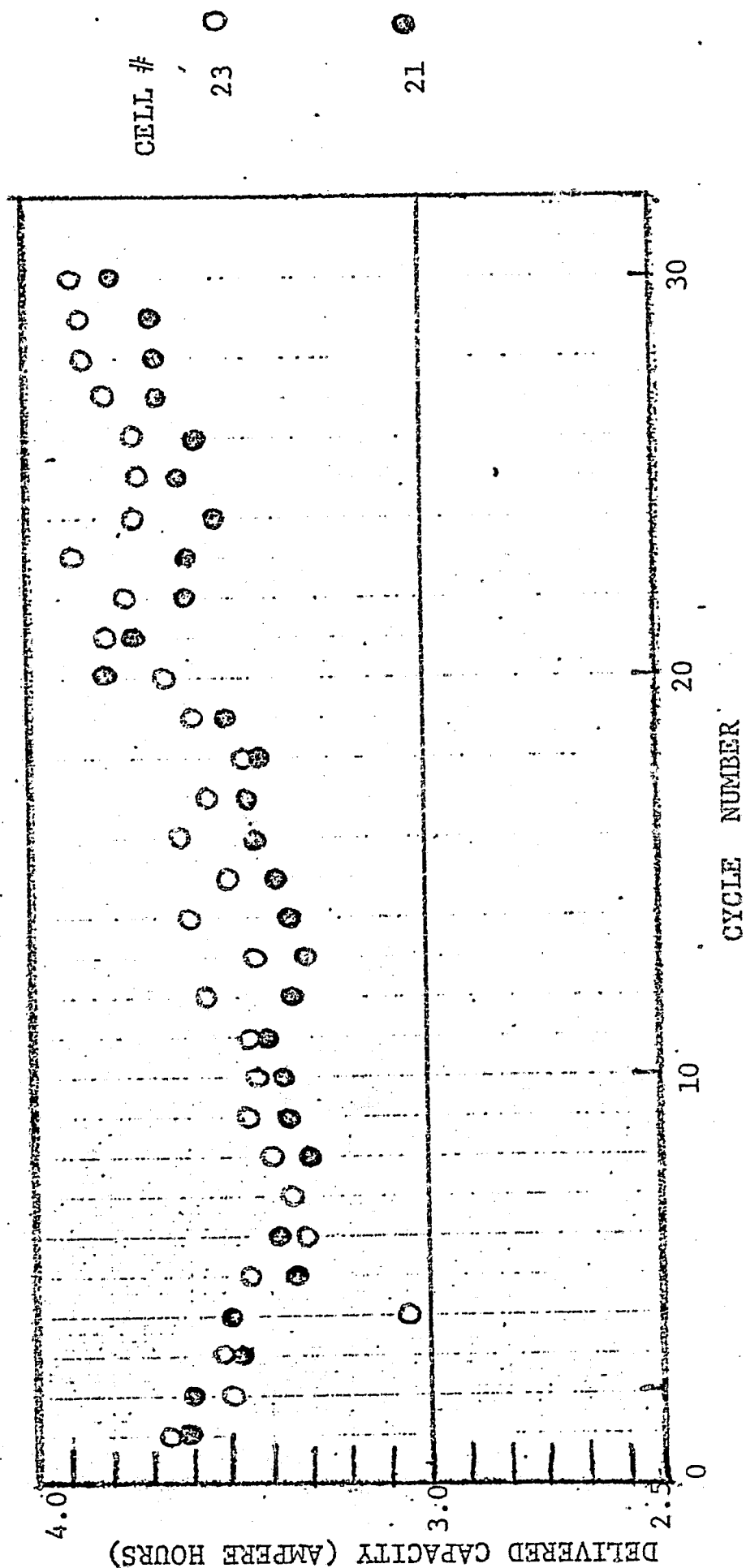


FIG. 4

AMPERE CAPAC. OF 17 PLATE FACTORIAL CELLS  
VERSUS  
CYCLE NUMBER

CELL DESIGN:

TYPE FT2140 SEPARATOR  
34 % KOH 80 % PORE FILL

STERILIZED

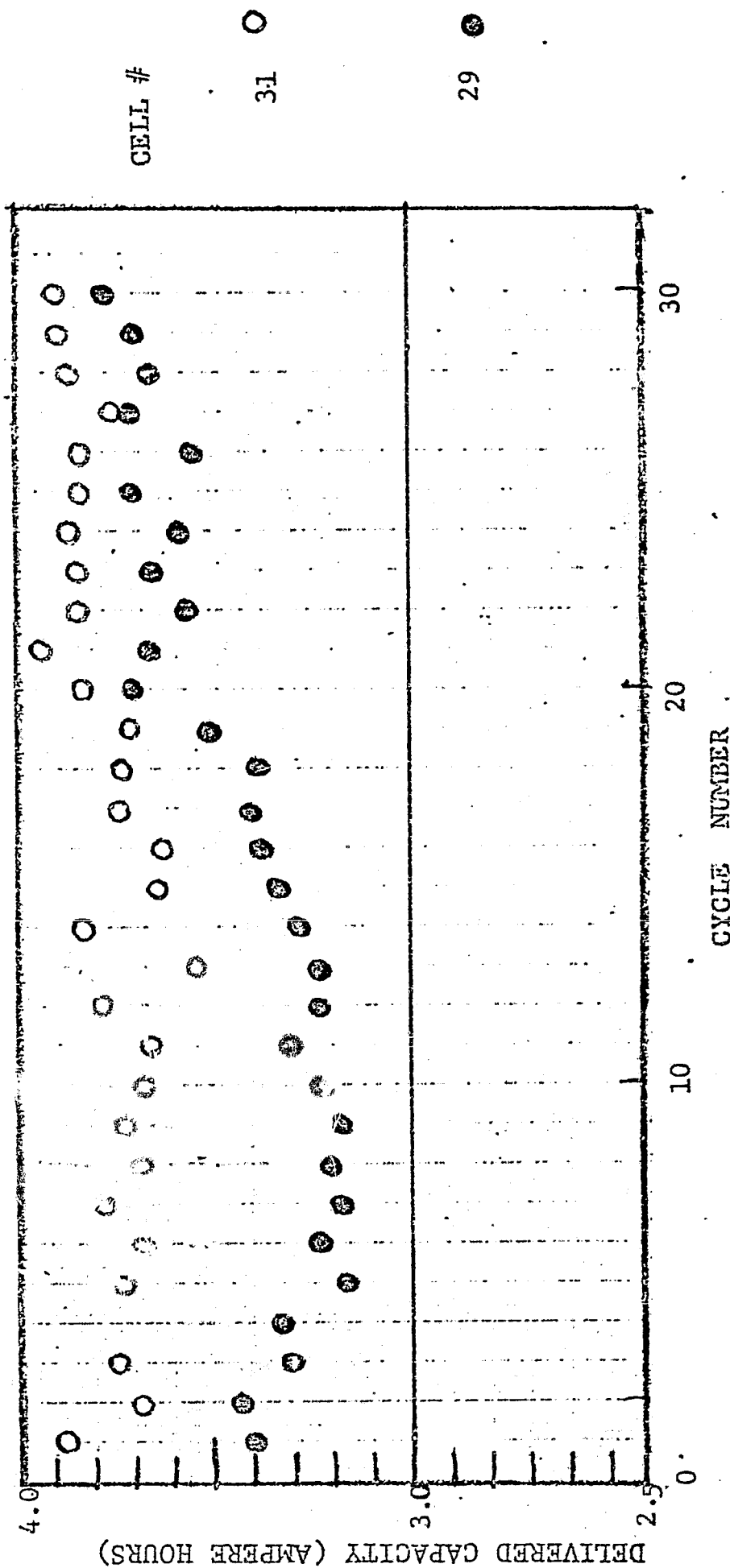


FIG. 5

END OF CHARGE VOLTAGE OF 17 PLATE FACTORIAL CELLS

VERSUS

CYCLE NUMBER

TYPE SEPARATOR FT2140 30 % KOH 70 % PORE FILL

STERILIZED

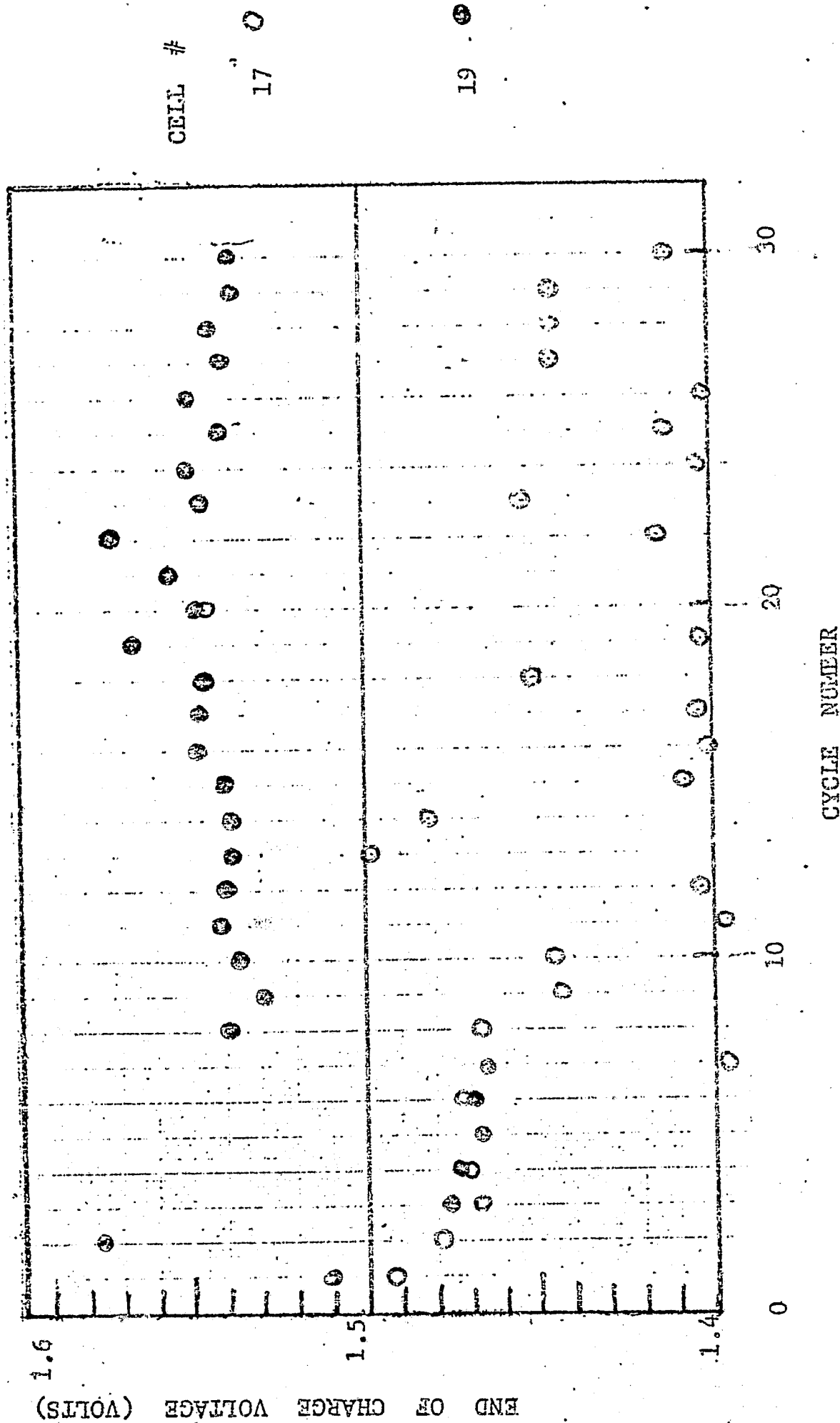


FIG. 6

END OF CHARGE VOLTAGE OF 17 PLATE FACTORIAL CELLS  
 VERSUS  
 CYCLE NUMBER  
 TYPE SEPARATOR FT2140 34 % KOH 70 % PORE FILL

STERILIZED

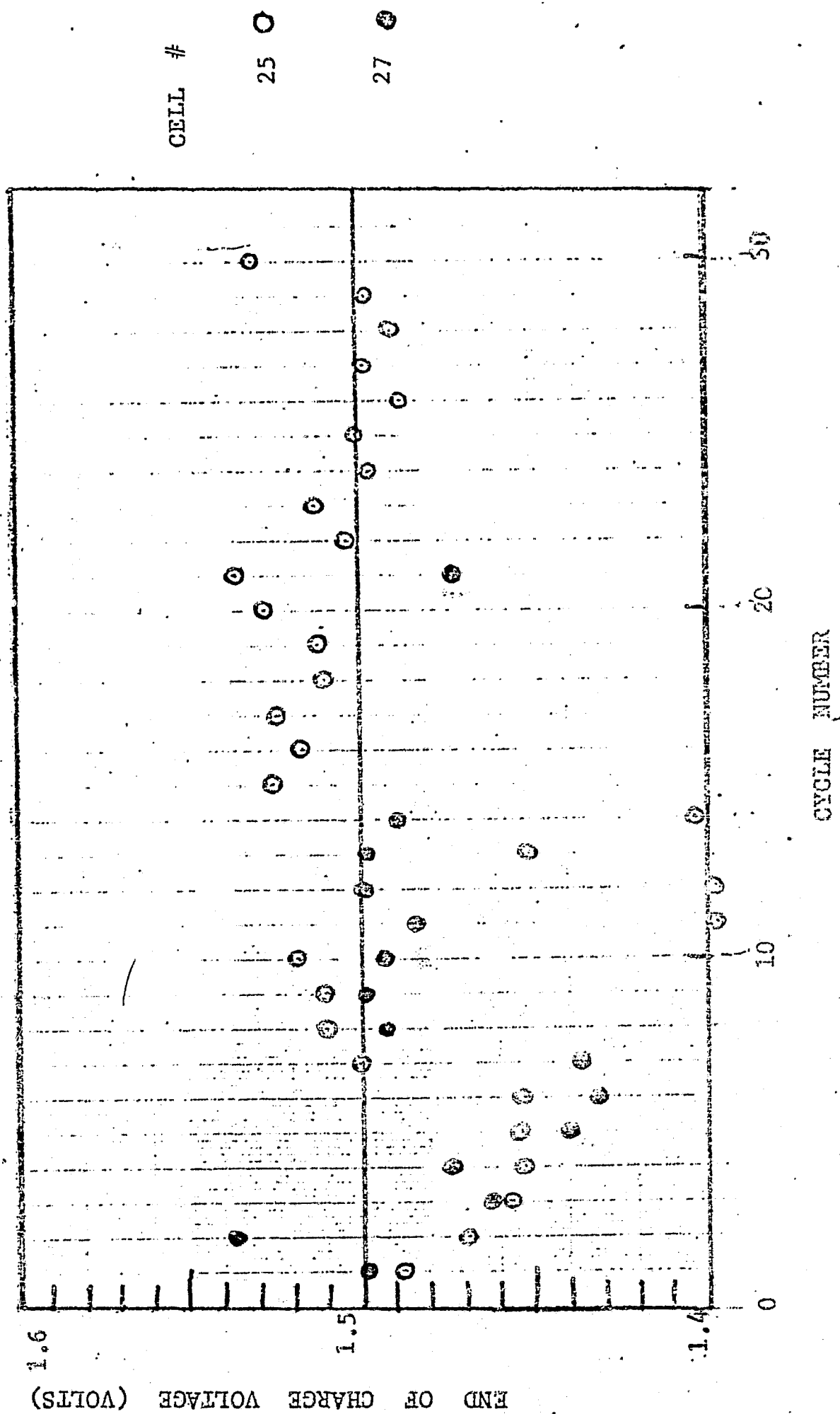


FIG. 7

END OF CHARGE VOLTAGE OF 17 PLATE FACTORIAL CELLS  
 VERSUS  
 CYCLE NUMBER  
 TYPE SEPARATOR FT2140 30 % KOH 80 % PORE FILL  
 STERILIZED

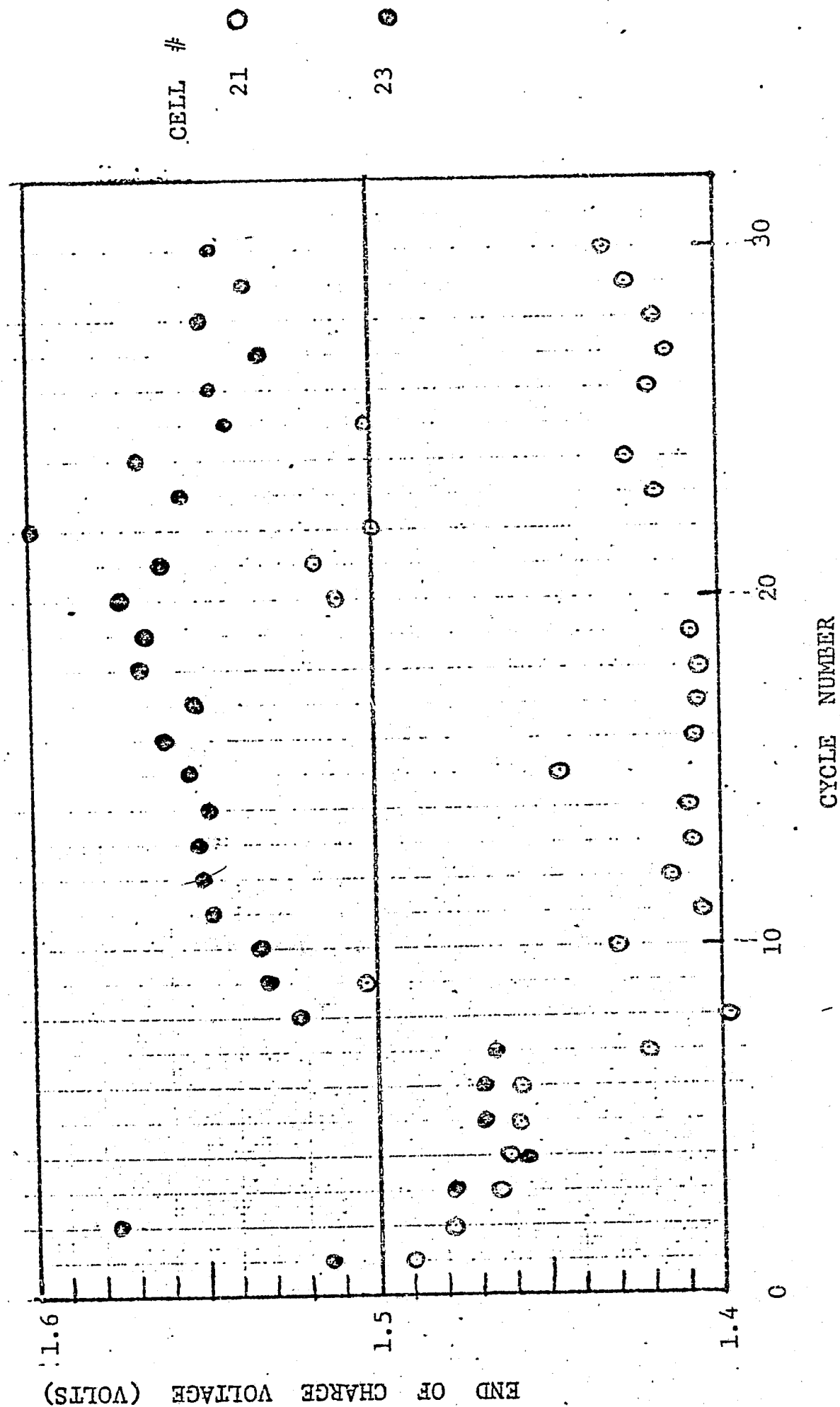


FIG. 8  
END OF CHARGE VOLTAGE OF 17 PLATE FACTORIAL CELLS

VERSUS  
CYCLE NUMBER  
TYPE SEPARATOR FT2140 34 % KOH 80 % PORE FILL

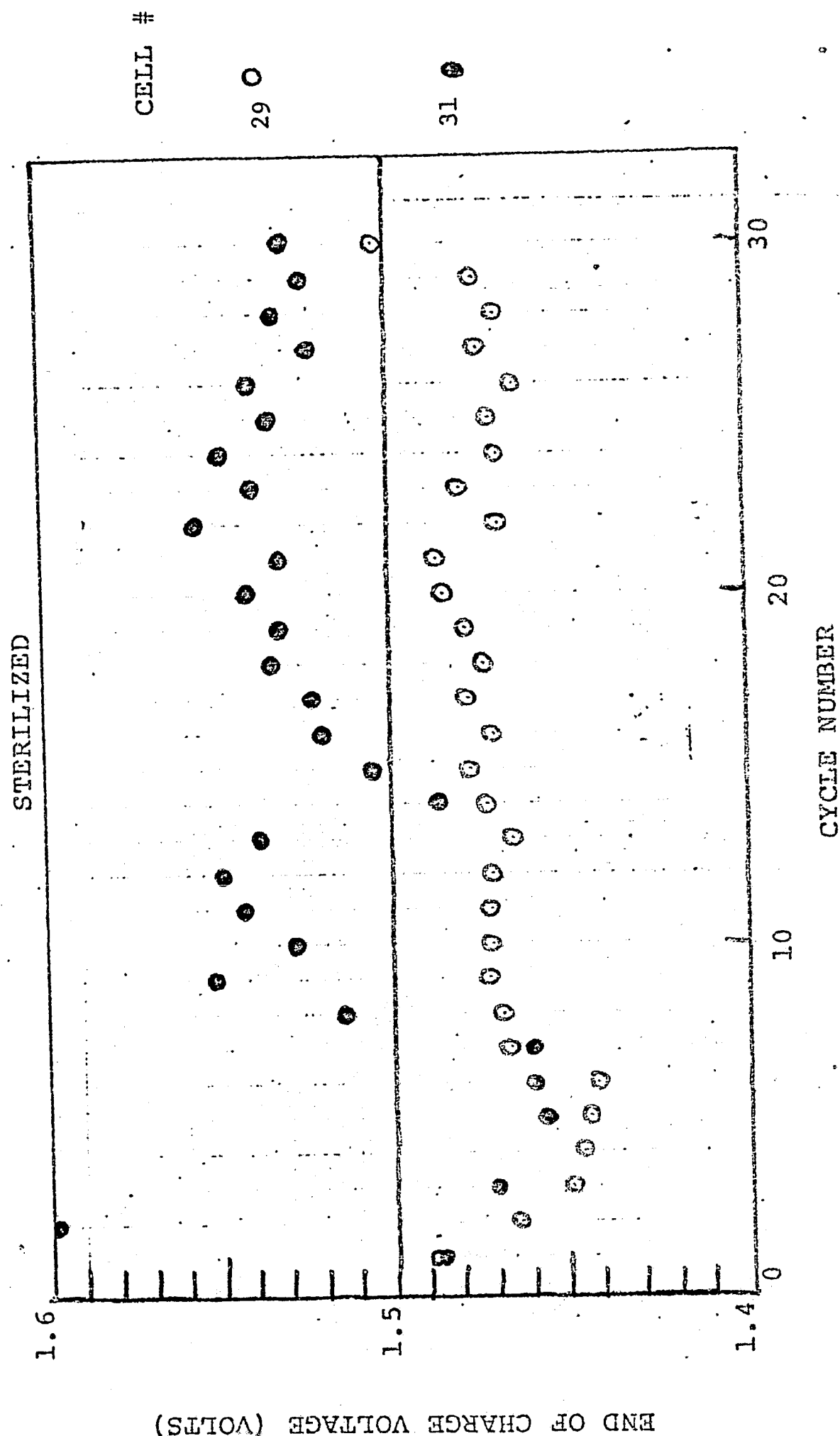


FIG. 9

END OF CHARGE RESISTANCE OF 17 PLATE FACTORIAL CELL  
VERSUS  
CYCLE NUMBER

TYPE SEPARATOR FT2140 30 % KOH 70 % PORE FILL

STERILIZED

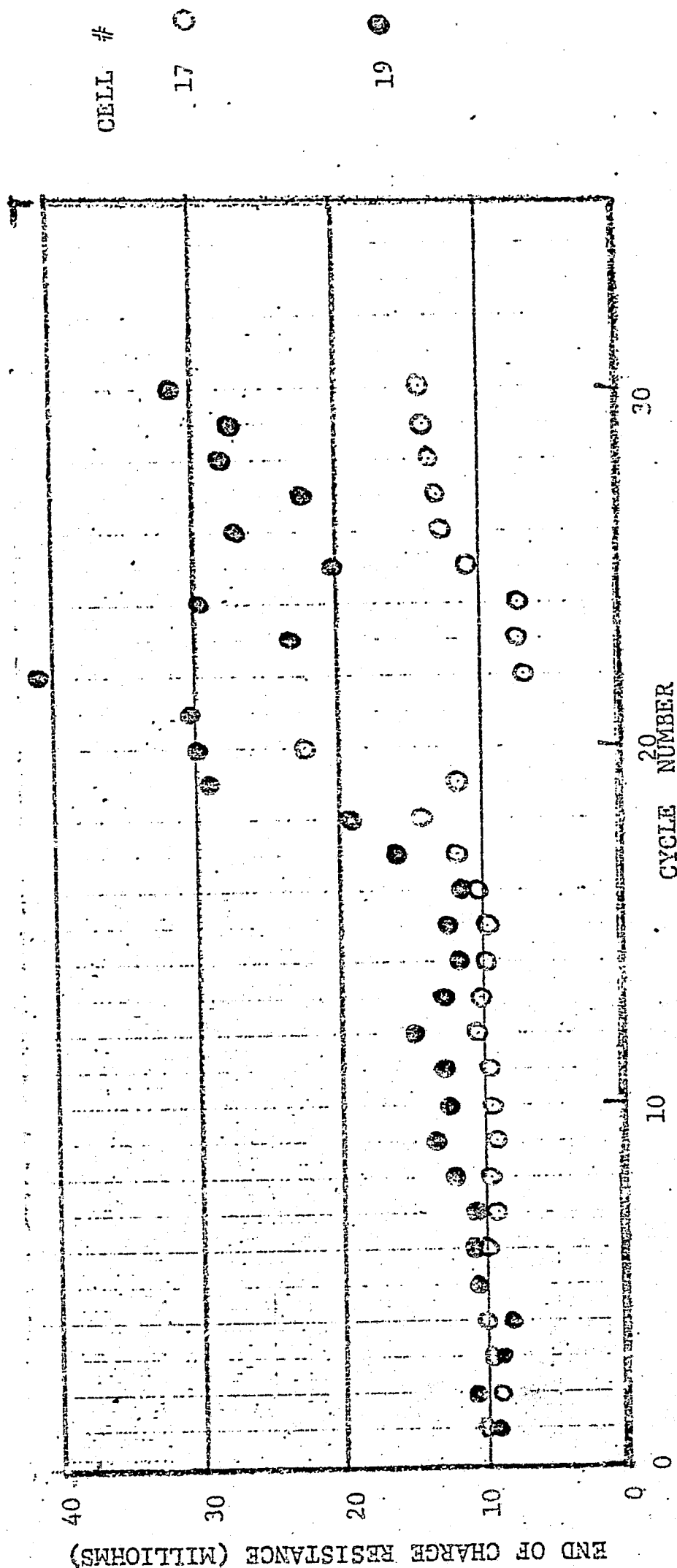




FIG. 10

END OF CHARGE RESISTANCE OF 17 PLATE FACTORIAL CELL  
VERSUS  
CYCLE NUMBER

TYPE SEPARATOR FT2140 34 % KOH 70 % FORT FILL

STERILIZED

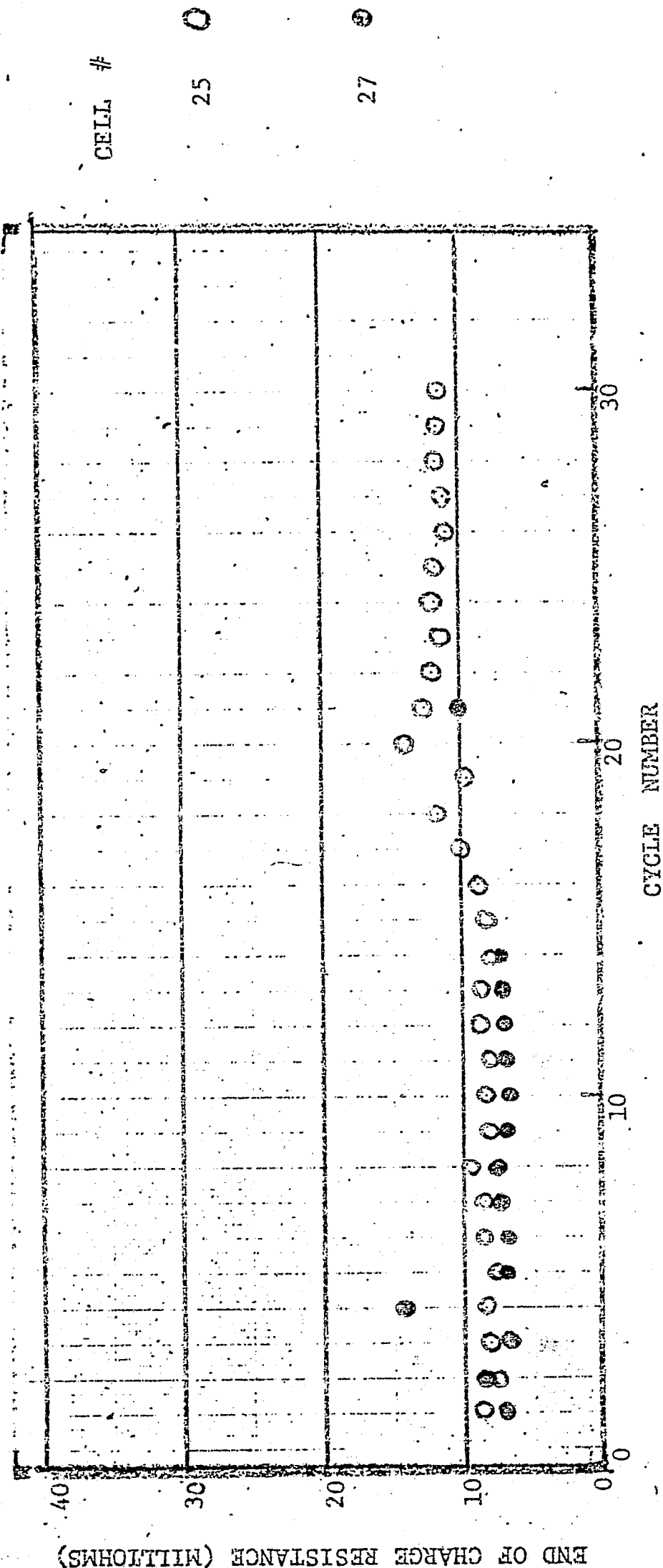


FIG. 11

END OF CHARGE RESISTANCE OF 17 PLATE FACTORIAL CELL  
VERSUS  
CYCLE NUMBER

TYPE SEPARATOR FT2140      30 % KOH      80 % FORE FILL

STERILIZED

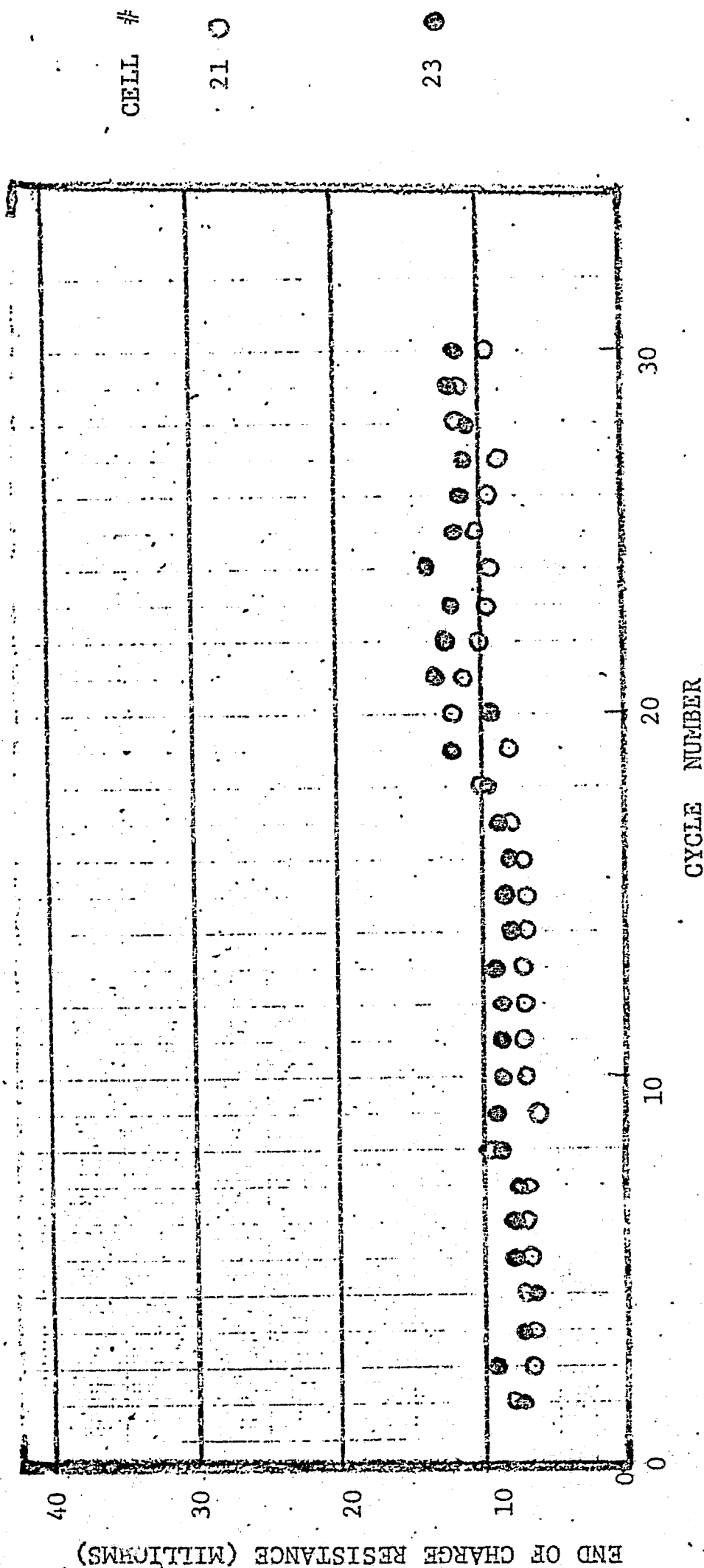


FIG. 12

END OF CHARGE RESISTANCE OF 17 PLATE FACTORIAL CELL  
VERSUS  
CYCLE NUMBER

TYPE SEPARATOR FT2140 34 % KOH 80 % PORE FILL

STERILIZED

